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EXPERIMENTAL INVESTIGATION ON MECHANICAL CHARACTERIZATION OF PAN CARBON

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ABSTRACT

The main factors that derive the use of composites are weight reduction, corrosion resistance, specific strength and stiffness, when compared to traditional materials or conventional materials. Characterization of each composite material is done before its application in product development. In the present work, the mechanical characteristic properties of Carbon as reinforcement will be studied. Laminates will be made with fabric of PAN, based Carbon and sample pieces are tested as per ASTM standards. The quality of laminates is verified and further tested for their mechanical properties like tensile, compression, flexure & ILSS. Fiber volume fraction is also determined from resin content and density.

KEYWORDS* : *Composites, Carbon Composites, Pan Carbo & ILSS

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Original Article

INTRODUCTION

The increasing use of composite materials in structural applications has returned a corresponding increase in like for experimental knowledge. The necessity to critically characterize composites has increased in recent years, thanks to the advanced nature of information needed for style thought. Composite technology development, check strategies' turned around measurement strength, stiffness beneath straightforward tension, compression and shear masses measurement of heat, thermal physical phenomenon, thermal diffusivity etc. of structural and thermal characteristics of composite structures, severally. Several of those check strategies won't characterize metals and be applied to fibre bolstered materials. The heterogeneous, anisotropic nature of fibre bolstered composite needs, but that any check methodology borrowed from silver technology is rigorously scrutinized before being directly applied to the characterization of composite materials.

In most engineering usage of fiber strengthened composite, laminate stacking pure mathematics is chosen, and such stiffness & strength are controlled by fiber modulus & strength, severally. Therefore, some matrix softening is accommodated in such applications, while not serious consequences. Significant matrix softening happens, however, thanks to transferring of load through the matrix to the fibers. The result's an amendment in failure mode from fiber dominated to matrix dominated. There are a unit, quite fifty thousand materials obtainable to engineers for the look and producing of product for varied applications. These materials vary from normal materials (e.g. copper, cast iron), that are obtainable for many hundred years, to the additional recently developed, advanced materials (composites, ceramics). Because of wide alternative of materials, the largest challenge is for

choice of a fabric and also the right choice of producing method for associate application. These materials were counting on their major characteristics (e.g. stiffness, strength, density, & melting temperature), whereas, carbon fibers is square measure of high interest in today's composite structures. Carbon fibers square measure is made up of organic precursor materials by a method of destructive distillation. They're a replacement breed of high-strength materials. Carbon fibers square measure is employed in composites with a light-weight matrix. Carbon fiber composites square measure is ideally suited to applications wherever strength, stiffness, lowers weight, and outstanding fatigue characteristics. They can also be employed in the occasion wherever extreme temperature, chemical resistance and high damping square measure necessary. The quality of the fibers depends on the composition and quality of the precursor fibers. It is made of textile, pitch or PAN (Poly Acrylo Nitrile) precursor fibers that are heat treated at temperatures of 1000–30000C and have markedly completely different in properties and structures; they contain ninety two to ninety nine percent carbon. The majority of carbon fibers are utilized in the region, and different structural applications are made of PAN fibers. Carbon fibers are made of varied varieties of pitch. Early carbon fibers were factory-made from rayon; but, these fibers are step by step phased out, because of their low carbon yield (20-25%), and their usually poorer mechanical properties compared to PAN and pitch-based carbon fibers. Carbon fiber consumption for chemical compound reinforcement is established at not up to five-hitter of the overall for all fibers. Specific uses of carbon fibers square measure high mechanical performance, fatigue behavior, high insulator & thermal physical phenomenon, lower density than glass fibers, low constant of friction and low constant of thermal enlargement. When plus the right organic compound, carbon fiber composites square measure one among the foremost corrosion resistant materials obtainable. They are doing not softener- soften with heat, permitting them to be utilized in such extreme temperature applications, as rocket nozzles square measure craft brakes. Their strength will increase with temperature in non-oxidizing atmosphere. PAN Carbon based, mostly phenolic resin Composites are bit by bit turning into the foremost advanced materials having higher properties to fulfill the Thermal needs. The partor missiles structures need high strength, light-weight weight and may face up to the high temperatures. Composite materials are accustomed to replace the standard materials, virtually in each field of application. PAN based, mostly phenolic resin composites are wide employed in air frame structures for part and missile application, thanks to their exotic properties reminiscent of low weight to high strength, and additionally withstanding higher temperatures. The properties of carbon fibers, equivalent to high stiffness, high durability, low weight, high chemical resistance, heat tolerance and low thermal growth, create them very hipping region, engineering, military, and motorsports, at the side of alternative competition sports. However, they're comparatively valuable when put up next with similar fibers, equivalent to glass fibers. Carbon fibers are composited with alternative atomic number 6 kind carbon-carbon composites that have a really high heat tolerance. Carbon fibers derived from Polyacrylonitrile (PAN) square measure turbostratic, whereas carbon fibers derived from mesophase pitch square measure graphitic, when heat treatment at temperatures surpassing 2200 °C. Turbostratic carbon fibers tend to possess high strength, whereas heat-treated mesophase-pitch-derived carbon fibers have high modulus of elasticity (high stiffness or resistance to extension underneath load) and high thermal physical phenomenon. Flexural takes a look at 2 totally different thicknesses with 2 different resins of bi-woven glass epoxy, carbon epoxy, and carbon epoxy specimens were compared with glass polyester, carbon polyester and carbon polyester resin tested and results recorded. The influence of specimen thickness and influence of rosin on the flexural properties were evaluated and it's found that the rise in thickness decreases the flexural properties, adore flexural strength & flexural modulus, because the thickness will increase the load carrying capability on the specimen. Flexural properties of Epoxy Glass, carbon and Carbon Laminates of 2mm and 4mm thicknesses with epoxy and Polyester rosin were successfully

concluded. The results of specimen thickness & resin on the flexural properties were evaluated, and it's found that the specimens strengthened with epoxy resin shows higher Flexural properties, as compared to the specimen strengthened with polyester resin. Finally, it will be complete that for same thickness and orientation, carbon fiber strengthened with epoxy resin provides higher flexural properties as compared to glass and carbon resin, forced with each epoxy and polyester resin underneath flexural loading conditions. Analysis indicates that Flexural strength is especially enthusiastic about the sort of resin used & thickness of laminated compound composites. The appropriate action temperature for oxide synthetic resin composite system was firm as 165°C, that is additionally, in accordance with reports addressing the organic compound vender for the E glass/phenolic system. At temperature higher than 165°C, there are no vital changes discovered in mechanical properties. As proved in the mechanical process and results obtained at longer action times, no further post curing treatment was needed. The mechanical properties of the composites didn't amend between eight and one hundred and fifty bars. This increase of pressure was caused solely by a small increase within the mechanical properties, suggesting that composites of optimum mechanical properties may well be created while not the employment of high pressures, comparable to autoclave techniques. Amendment in action conditions, like temperature, period and pressure did lead to any vital amendment to the fiber/matrix volume fractions, density or porousness of the composites, that expedited the comparison of the mechanical properties in composite laminates, created underneath in completely different action conditions. Additionally, the composite laminates had medium fiber volume fractions (40-50 %), of course from the standard impregnation technique applied. The results obtained from this study may be used for processes (autoclave etc.), nevertheless hot pressing for sensible functions. Phenolic resin is employed as a matrix material attributable to its wonderful thermal properties. Phenoplast changed by Di-amine exhibited high flexural and impact strength. For top temperature applications in part, carbon phenolics are used as ablative materials attributable to their low erosion. Phenolic systems generate water as a reaction by-product, via condensation reactions set throughout at elevated temperatures. Within the fabrication of fiber strengthened phenol matrix composites, volatile management is crucial in manufacturing void-free quality laminates. Bidirectional carbon fiber and bolstered epoxy composites show higher mechanical properties, i.e., strength, inhume laminate shear strength, flexural strength and impact strength, apart from that of hardness, wherever the values for brief carbon fiber bolstered epoxy composites square measure over that for biface composites. Increase in values for biface carbon fibers is also due to the uniform fiber orientation in all the told directions. The plasticity of carbon fiber bolstered composite is over the opposite composites. PAN based carbon fibers, however, are evaluated as various reinforcement for nozzle insulators for over fifteen years. Thermal conduction, an important performance property, was found to be adjustable by variable the carbonisation temperature. By dropping carbonisation temperature below 1400°C, PAN-based carbon fiber conduction values is reduced to concerning common fraction of normal PAN-based fibers. Though there's heaps of literature accessible on the composite materials for top temperature missions, still there's demand to reinforce their performance to form the regional missions, reliable and safer. Any cracks generated, where loading can deteriorate the performance of the fabric and can cause a harmful failure. Thermal characterization of extreme temperature structures like heat shields, thermal protection system for rocket casings and nozzles is incredibly vital in composite materials for applications.

Aim of the Project

Aim of this project is to characterize the PAN Carbon Phenolic. PAN carbon phenolic composites are used as ablative liners requiring good thermal and mechanical properties. However, mechanical properties of these materials have to be determined, since in applications, the minimum mechanical requirements have to be met. The mechanical properties

of the materials will be determined. Mechanical properties such as Tensile, Compression, Flexure and ILSS of these composite materials are to be found.

MATERIALS AND METHODS

Materials used in this work are PAN Carbon fabric and Phenolic resin. Material properties are usually determined by mechanical and physical tests, under controlled laboratory conditions or from manufacturer or supplier.

Figure 1: Liquid Phenolic Resin

The Phenolic Resin, which we used is a RESOLE type; purchased from ABR Organics. It is highly suitable for prepreging of fabrics, which finds application in ablative liners for aerospace & defense applications. Specification of Phenolic resin (as received) used in the study is as per Table 1.

Table 1: Phenolic Resin Parameters

Type	Resole
Appearance	Brown viscose liquid
Point of trouble, ml	6.0 - 12.0
Viscosity @300C	150 - 350 CpS
Volatile content, by weight%	32 - 38
Solid resin content, by weight%	60 - 65
Specific gravity	1.12 – 1.16

PAN Carbon Fabric is in the largest production and best used in volume. Carbon fiber is the strongest fiber in strength & modulus among the other fibers available. It is a structural and ablative material. The specifications of this fabric are as per the Table 2.

Table 2: PAN Carbon Fabric Parameters

Parameters	Nominal Value
Yarn T 300	3000 filament
Thickness, mm	0.41 to 0.45
Warp	24 ± 1 tows/in.
Weft	24 ± 1 tows/in.
Area/Density	370 – 400 grams/m^2
Type of weave	8-H satin weave
Width	1 meter
Breaking strength	Warp/Weft 8 to 9 kg/mm width
Density, g/cc	1.7 to 1.8

TOOLS & EQUIPMENT

- Rubber Squeegees are excellent for forcing resin through high performance fabrics without the fear of snagging or distorting costly fibers.
- Scissors enables to easily cut fabric that is flat on a table.
- Top and bottom mould plates for stacking of layers.
- Spacer blocks are placed in between the top and bottom mould plates to provide particular thickness after curing. These spacers allow the excess resin and vacuum to flow out from the pre-cured laminate.
- Rollers are straight across the width of the head and provide excellent air relief between the layers while stacking.
- Diamond wheel cutter is used for cutting the cured laminate for specific dimensions or specimens.

Processing a Laminate

Phenolic resin was conditioned and the point of trouble reached 7.2 ml on the day of applying resin.

Impregnation

Fabric was weighed as per the required laminate dimensions. Phenolic resin was weighed equal to that of the fabric. A prepreg of the fabric was prepared by applying the wet resin on the fabric using a rubber squeegees and kept to conditioning a week to reduce volatiles and achieve proper tacking.

Figure 3: Applying Phenolic Resin on Fabric

These are the prepreg values on the day of layup:

Table 3: Prepeg Parameters

Prepreg Parameter	Silicaphenolic	PAN Carbon phenolic
Volatile content, by wt %	7.28	7.49
Solid resin content, by wt %	43.23	48.77
Fiber content, by wt %	47.16	41.97
Chang's index, ml	21.1	8.5
Layer thickness, mm	0.8	0.5

The processing steps in the lay-up: Pre-preg is cut into 320*320mm dimensions using templates. A release agent is applied to the mold and spacers. Prepreg is placed directly on the bottom mold surface.

Using a roller, the prepreg is pressed to remove air bubbles between two layers. Subsequently, the prepreg layers are stacked until a required thickness is built up. 5 no. of layers are stacked for Silica Phenolic to achieve the required

thickness. 8 no. of layers are stacked for PAN Carbon Phenolic to achieve the required thickness. The ply layers are covered with the top mould.

During Consolidation, due to the application of pressure, fibers are compacted together.

Consolidation pressure is applied during layup with the roller. The mould is then closed, forcing the material to flow and consolidate. Mould, when placed in the hydraulic press, bolsters add pressure while curing. The pressure is applied during the curing process mentioned in the Table 5.5; with high temperature ramp-and-hold period is to afford laminate consolidation. Pressure is applied before 1200C through hydraulics of about 10 tons for consolidation. Consolidation of prepreg plies in the final component is done during the curing process.

Consolidation pressure is applied to attain the desired physical properties of the resin matrix and good mechanical properties for the composite. These loads produce corresponding increase in the vertical effective stress. Once the consolidation pressure is applied, residual volatiles, if any, are locked in and are unable to escape.

Figure 4: Consolidating while Layup Process

SOLIDIFICATION CURING

The final step is solidification. Complex heat transfer takes place as the material cures and solidifies. Solidification starts at the temperature of 1200C for phenolic based composite.

Figure 5: Mould of Laminate in Hydraulic Press

Mould assembly is loaded in hydraulic press for curing, as per the curing cycle shown in Figure 5

Table 4: Curing Cycle

Temperature (^{0}C)	Time (hours)	Pressure (tons)
90	2	-
120	2	10
165	2 ½	10
Cooling	Press switch off	Natural cooling

Heat is applied during processing to expedite the cure rate of resin, higher the cure temperature, faster the cross-linking process. After cooling mould is dismantled and laminates are extracted from the mould.

Figure 6: Silica Phenolic Laminate

EXPERIMENTS AND RESULTS

Specimens for physical, mechanical and thermal tests were cut using a diamond wheel cutter, as per required dimensions. Physical, mechanical properties of a material are the basic design data in many, if not most, applications. The physical, mechanical properties are tested as per ASTM standards. The performance of an engineering material is judged by its properties, behavior under tension, compression, shear and other static or dynamic loading conditions in both normal and adverse test environments. This information is essential for selecting the proper material in a given application, as well as designing a structure with the selected material.

Table 5: ASTM Standards of Specimens

Test Performed	Specimen Size
Thermal Diffusivity	320*320*3mm (laminate as trimmed)
Tensile	250*25*3mm
Compression	120*12.5*3mm
Flexural	110*12.7*3mm
ILSS	60*10*3mm
Other tests samples	20*20*3mm

Sample Characterization

The density of the test specimens is then determined as per ASTM D792. The specimens are then placed in an appropriate dissolution medium and heated/refluxed, until all of the organic material is dissolved. The resulting solution is then filtered and the amount of remaining fiber is determined. From the specimen density, constituent densities, and the fiber content, the reinforcement content, matrix content, and void content can then be determined. ASTM D3171 standard was used for burn-off tests that are to see the fiber volume fraction of the composites. Five specimens of 25mm x 25mm were ready from totally different made up laminate panels.

$Vf = (Mf/Mc) \times 100 \times (\rho c/ \rho f)$

Where:

Vf = volume percent of reinforcement in the specimen Mf =final mass of specimen after combustion

Mc =initial mass of specimen after combustion

ρc =□density of the composite specimen

Tensile Test

Tensile properties corresponding to strength and tensile modulus of composite laminates area unit are determined by static tension tests, in accordance with ASTM D3039. Tensile tests turn out a stress-strain diagram that is employed to see tensile modulus. In this project, Instron Universal Testing Machine (UTM) with loading rate of 2 mm/min was used.

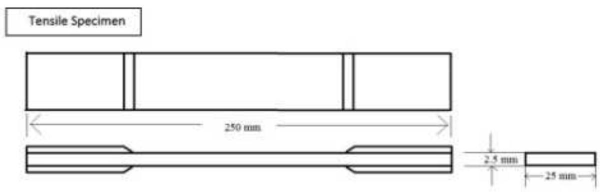

Figure 7: Testing Specimen of Tensile Test

Flexure Test

Flexural strength is decided by ASTM D790, it measures the force needed to bend a beam underneath three point loading conditions. The information is commonly accustomed to choose materials for elements that may support masses while not flexing. Flexural modulus is employed as a sign of a material's stiffness, once flexed. During this check, the composite beam specimen of rectangular cross section is loaded in 3-point bending mode, an outsized span to thickness quantitative relation is suggested. Accordingly, the specimen was made to the dimension of 8mm*16mm*48mm

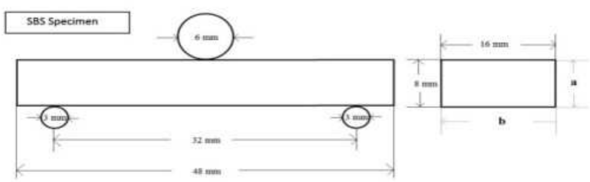

Figure 8: Testing Specimen of Short Beam Shear

INTERLAMINAR SHEAR STRENGTH

Short Beam Shear ASTM D2344 refers to the shear stress developed at the specimen mid-plane at failure. To calculate the short-beam strength, most loads throughout take a look at, and therefore the specimen breadth and thickness values should be employed in the equation provided by the quality. Specimen size for ILSS is 10mm wide and 60mm long. Span: thickness magnitude relation for ILSS is 5:1.

Figure 9: Flexure and ILSS Specimens

Interlaminar shear failure is most readily seen in the three-point bending of short beams, a method commonly used to measure what is usually referred to as the interlaminar shear strength, or ILSS.

COMPRESSION TEST

Compressive property was measured as per ASTM D695. It describes the behavior of a fabric, once it's subjected to a compressive load. Loading is at a comparatively low and uniform rate. Specimen size for compressive check is taken as twelve.5mm wide and 120mm long.

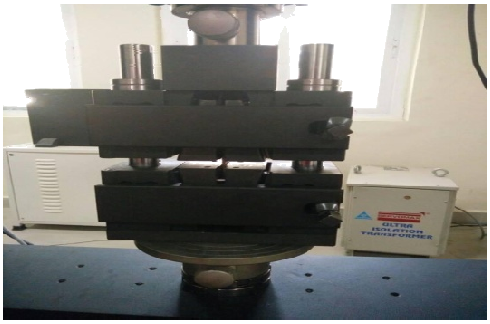

Figure 10: Compression test Fixtures

RESULTS & DISCUSSIONS

Resin content and density for the PAN Carbon Phenolic composite material is:

Table 6: Composite Properties of Resin Content and Density

Properties	Experimental Value
Resin content, (by weight %)	29.55%
Density (g/cc)	1.508

From the table, Wm=0.29 and from equation of rule of mixture volume fraction of fiber is calculated i.e.

Wf = 0.71

From weight fraction of fiber, fiber volume fraction is calculated as Vf = 0.603

Void content will be $\boldsymbol{v_{\text{void}} = 4.071\%}$ Therefore; void content in PAN Carbon Phenolic is 4.071%

The mechanical and thermal properties of PAN Carbon phenolic composite are determined at room temperature.

Results of mechanical tests on PAN Carbon Phenolic specimens are;

Table 7: Mechanical Properties of PAN Carbon Phenolic

Properties	Experimental Value
Tensile strength (MPa)	640
Tensile modulus (GPa)	78.33
Compressive strength(MPa)	338
Flexural strength(MPa)	338
ILSS (MPa)	37.03
Fiber volume fraction (Vf) (%)	0.603

The tensile properties of PAN Carbon Phenolic composites are good. PAN Carbon fabric has high strength properties. The flexural strength properties are lower than the tensile strength.

The compressive strength properties are satisfactory. The failed specimen of compression test of PAN Carbon Phenolic is shown in figure.

Figure 11: PAN Carbon Phenolic Specimen of Compression Test

The ILSS property may be further improved by increasing the resin content and increasing the affinity of fabric with matrix.

A material with high modulus of elasticity will make a stiff structure. The stiffness of carbon fiber is the fact that it tends to be more brittle. When it fails, it tends to fail without showing much strain or deformation as a catastrophic failure.

Diffusivity property of carbon phenolic is good. Carbon Fiber is resistant to elevated temperatures, it either have a melting point and have been used for protective clothing or the fabric used near fire. Carbon is used to make protective fire fighting or welding blankets or clothing.

The PAN Carbon fabric, being thermally conductive has resulted in a composite with higher thermal conductivity. PAN Carbon Phenolics can be used in applications require high mechanical properties with high temperature resistance.

Porosity level in the PAN Carbon Phenolic composite material is 4.071%, and pores in the specimen reduces the material's mechanical and thermal properties. Materials with more than 5% porosity level have less strength. We can assume that presence of pores might have led to lower flexural strength than tensile strength.

The thermal conductivity & specific heat properties of the composite are assumed to remain constant with increasing temperature. Porosity level has dramatic effect on Thermal properties; pores are filled with low thermal gases, which overall reduces the properties of composite. PAN Carbon Phenolic degrades by melting and charring.

CONCLUSIONS

- Pan carbon fabrics were impregnated with resoles type of phenolic resin. Using hand lay-up technique, prepreg of 320*320mm dimensions were stacked to get the laminates of 3mm thick after compression molding. Specimens are cut as per ASTM testing dimensions. Mechanical properties were successfully tested.
- The Mechanical properties evaluated were Tensile strength, tensile modulus, Compressive strength, Flexural strength and ILSS. The results obtained showed that PAN Carbon Phenolic bidirectional composite has average values of 640MPa, 78.33GPa, 338MPa, 338MPa, 37.06MPa for Tensile strength, tensile modulus, Compressive strength, Flexural strength and ILSS, respectively.
- Bidirectional fabric transfers the load from one directional fiber to the fibers of other direction. The weave pattern 8HS gives more flexibility to the fabric to drape.
- PAN Carbon Phenolic has excellent mechanical properties. In this, material flexural strength was lower than tensile strength, possibly due to presence of 4.071% porosity.

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